Synthesis of Crosslinkable Hole Transporting Polymers for Organic Light Emitting Diodes (OLEDs)

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Synthesis of Crosslinkable Hole Transporting Polymers for Organic Light Emitting Diodes (OLEDs)

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LIST OF ABBREVIATIONS

OLED	Organic Light Emitting Diode
HTL	Hole Transport Layer
BCB	Benzocyclobutene
TFVE	Trifluorovinyl Ether
OXT	Oxetane

ABSTRACT

As a result of intensive research over the course of the past decade and beyond, great strides have been made in developing organic light emitting diodes (OLEDs) for display and solid-state lighting applications. One of the key remaining obstacles to OLED production is the difficulty of fabricating the multi-layer architecture required for high-performance devices. Vacuum deposition, the conventional fabrication approach is expensive and is not readily scalable, while the main alternative approach - solution processing is hindered by inter-layer incompatibilities. A promising answer to the latter issue involves rendering each layer insoluble after deposition by inducing crosslinking, thus making the material compatible with the next layer deposited above it. The goal of this project was the preparation of novel crosslinkable materials for applications as the hole-transport layer (HTL) in OLEDs. A series of copolymers was prepared, integrating a hole transport group on one chain and a crosslinker on the other. The crosslinking groups studied were benzocyclobutene (BCB), oxetane (OXT), and trifluorovinyl ether (TFVE). Thin films of the target materials were prepared, processed to induce crosslinking, and their solvent resistance was evaluated. It was found that materials incorporating BCB and OXT groups exhibited effective solvent resistance, even after relatively short processing times.

INTRODUCTION

Light emitting diodes (LEDs) have been one of the most intensively studied fields of materials science during the past half century, as a result of which, great strides have been made in the understanding of the principles underlying their operation and in their implementation into a wide range of devices.[1] While initial research on LEDs focused exclusively on inorganic materials, the discovery of efficient luminescence in organic compounds (originally in anthracene) spawned the new subfield of organic light emitting diodes (OLEDs).[2] The advantages presented by OLEDs over their inorganic analogs, most notably their significantly lower cost, greater physical flexibility, and high tunability were readily seen and generated much interest in the new field.[2] Nevertheless initial efforts to develop viable OLEDs were greatly hindered by certain deficiencies characteristic to these devices, most importantly, their low efficiency.

The problem of efficiency was greatly reduced by the transition from single layer to multilayer devices. The earliest OLEDs consisted of a single organic electroluminescent layer sandwiched between two electrodes. The efficiency of these first generation devices was invariably quite low, a representative example being the first thin film OLED described by Tang and VanSlyke in 1987, which had an external quantum efficiency (the ratio of the radiative energy to the energy input) of ca. 1%.[3] New devices containing an emitting layer flanked by additional layers with suitable hole and/or electron transporting properties proved to be capable of significantly improving the external quantum efficiency.[4] The basic structure of such multilayer devices usually consists of a transparent anode (usually Indium Tin Oxide (ITO)) deposited on a glass

substrate, followed by a hole transport layer (HTL), an emitting layer (EML), an electron transport layer (ETL), and finally the cathode as depicted in Figure 1.[5]

The sequential deposition of the distinct layers is a problematic aspect of OLED construction. Two general methods are used for the addition of the layers in modern state of the art devices[4]: 1) vacuum deposition[6, 7] and 2) solution processing[8-10]. Vacuum deposition involves the sequential sublimation of each layer in a deposition chamber. This method is well-suited towards the fabrication of small devices and offers a very high level of control of the deposition of each individual layer. However, vacuum deposition is difficult to extend to large surface areas and is associated with a high fabrication cost. Furthermore, not all materials are compatible with vacuum deposition: candidate materials must be both thermally stable and sufficiently volatile for sublimation to be practical, which generally restricts this approach to small molecules.[4]

Solution processing involves the preparation of a solution containing the material constituting one layer and its deposition unto a substrate through spin-coating, inkjet printing, or other solution-based techniques.[4] This method can be less expensive than vacuum deposition and is more amenable to larger surfaces. Furthermore, solution processing is compatible with a much greater range of materials, including small molecules, polymers, as well as dendrimers. However, in contrast to vacuum deposition, solution processing offers a significantly diminished level of control over the deposition of each layer. This factor, in conjunction with the difficulty of purifying commonly used materials and the impurities introduced by the solvents utilized generally results in reduced performance for solution-processed devices.[11]

Another key problem encountered in the deposition of solution processed layers is that the previous layer can be dissolved by the solvent in which the new layer is processed. Three approaches have been developed to address this problem[4]:

a. usage of orthogonal solvents for successive layers,
b. change of polarity of a precursor in a certain layer
c. usage of crosslinkable polymers.

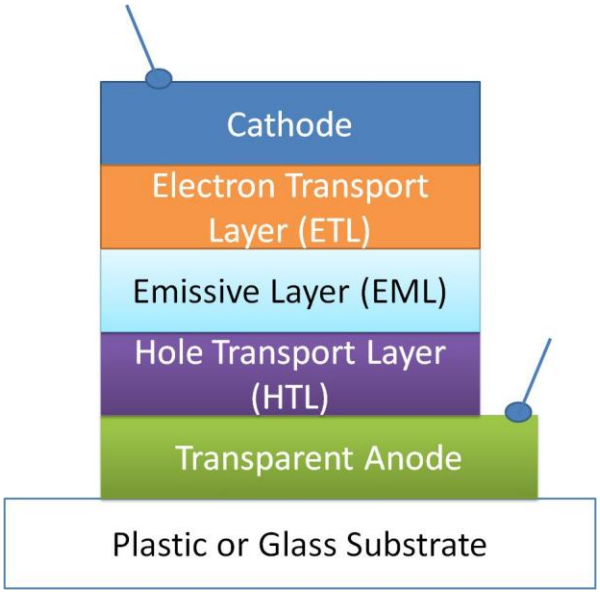


Figure 1.

The first method involves choosing the materials making up the layers of a device in such a way that the solvent in which one layer can be processed will not dissolve the layer immediately below it. The practicality of the orthogonality approach for multilayer OLED fabrication has in fact been demonstrated by multiple groups, by using a combination of materials soluble in water, alcohols, and other organic solvents.[12, 13] Nevertheless, only a limited range of materials can readily be made compatible with this

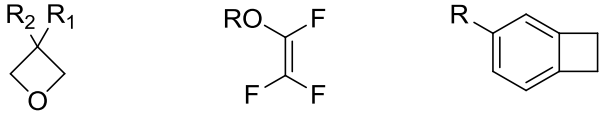
solubility criterion, greatly diminishing the applicability of this method. The second approach has also proven to be of rather low importance, due to the difficulty of finding classes of materials, the polarity of which can be altered after deposition.[4]

The third method involves first depositing a polymer in solution (e.g. by spin coating), then inducing crosslinking (the formation of covalent bonds between polymer chains) by exposing the material to ultraviolet irradiation or to high temperatures. The effect of the crosslinking is to render the layer affected insoluble, thus making it compatible with a solution processed layer deposited above it. As a result, much work is currently being dedicated to the development of crosslinkable materials that display the physical properties requisite of a specific OLED layer.[4]

Three crosslinking groups reported to have been incorporated in OLED materials are: the UV and thermally crosslinkable oxetane group[14], and the thermally crosslinkable benzocyclobutene (BCB)[9] and the trifluorovinyl ether (TFVE) groups[10] (see scheme 1).

Scheme 1

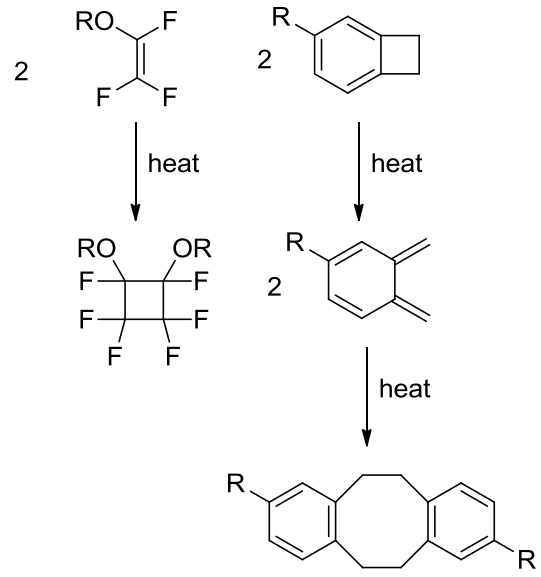
Oxetane Trifluorovinyl Ether: Benzocyclobutene:



Of these three groups, only benzocyclobutene and trifluorovinyl ether have been reported as having been used as the crosslinking groups in copolymers having applications in OLEDs. The crosslinking products for the two groups are indicated below: [9,10]

Scheme 2

Trifluorovinyl Ether: Benzocyclobutene:



Crosslinkable copolymers implementing both the benzocyclobutene and trifluorovinyl ether groups have been integrated in OLEDs.[9,10] The best result reported for such devices was a maximum external quantum efficiency of more than 10% measured by Fréchet et al. for an OLED containing a BCB-based crosslinked copolymer as the hole transport layer (HTL).[9] Such results suggest that the use of crosslinkable polymers represents a viable route for addressing the fabrication problems of state-of-the-art multilayer OLEDs. Nevertheless two crucial outstanding issues remain: 1) The processing times required for crosslinking can be excessive for practical applications. For example, for the polymer by Fréchet et al. mentioned above, effective crosslinking was achieved after 4 hours of heating at 200^{o}C, which was preceded by two hours of annealing at 170 ^{o}C.[9] 2) Another significant problem is that of efficiency. Even the highest efficiency reported for a device containing a solution processed crosslinked layer is significantly lower than the efficiency of state-of-the-art vacuum deposited devices.

Furthermore, the devices incorporating a crosslinked layer frequently only contain one crosslinked or solution-processed layer (usually the HTL). Imposing the requirement that all or most layers consist of crosslinked polymers can be expected to further increase the challenge of obtaining high efficiency devices.

The aim of the current project was to explore possible solutions to the first problem described above. The specific goal was the preparation of a series of hole-transporting copolymers capable of undergoing effective crosslinking after short processing times. Experimental objectives included the synthesis of the desired copolymers as well as the optimization of processing conditions in order to minimize the length of time required to induce a significant level of crosslinking.

METHODS

The general procedure of the project can be subdivided into the following three parts:

I. Synthesis of Copolymers

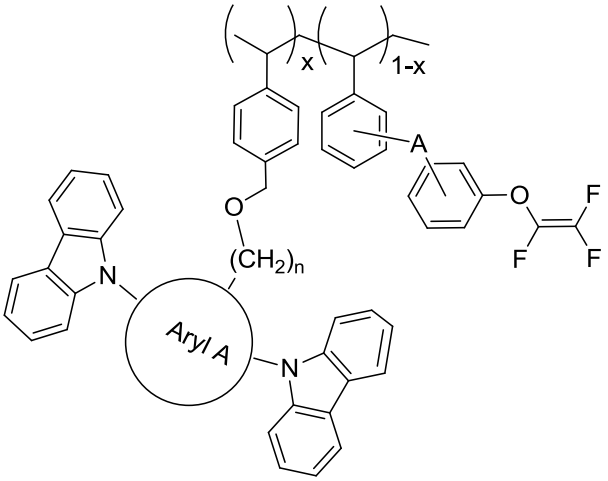
II. Thermal Crosslinking of BCB and TFVE Copolymers

III. Rapid Thermal Processing (RTP) of BCB, TFVE, and OXT Copolymers

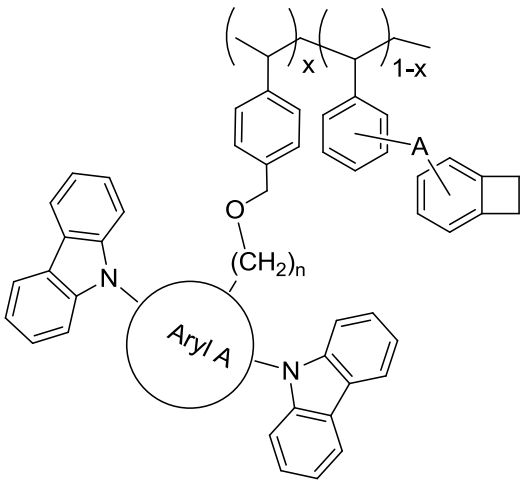
I. Synthesis

The first part of the project involved the synthesis, purification, and characterization of crosslinkable copolymers with suitable hole transporting properties, intended for the hole transport layer (HTL) of OLEDs. Specifically, the synthetic target involved the creation of a series of copolymers containing two chains: one containing a thermally crosslinkable group (BCB, TFVE, or OXT) and the other a group known to display suitable hole transporting properties, to be used as the hole transport layer in OLEDs. Preparation of the copolymers required first synthesizing the constituent monomers, then effecting the final polymerization. Due to the significant number of steps required to arrive at any of the terminal material from the available starting compounds, this component of the project was the most time intensive.

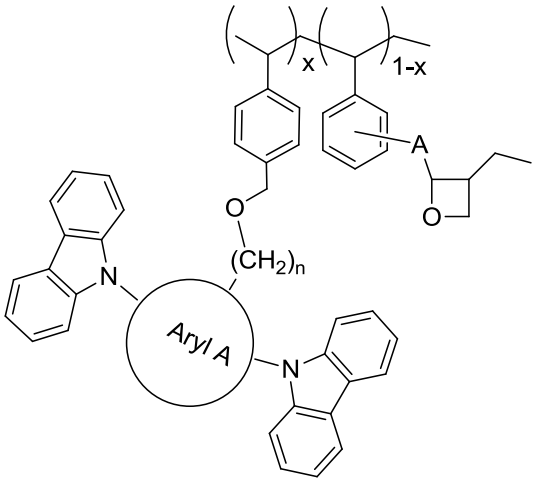
Scheme 3



IC-II-32/IC-II-53



IC-II-30



IC-II-99

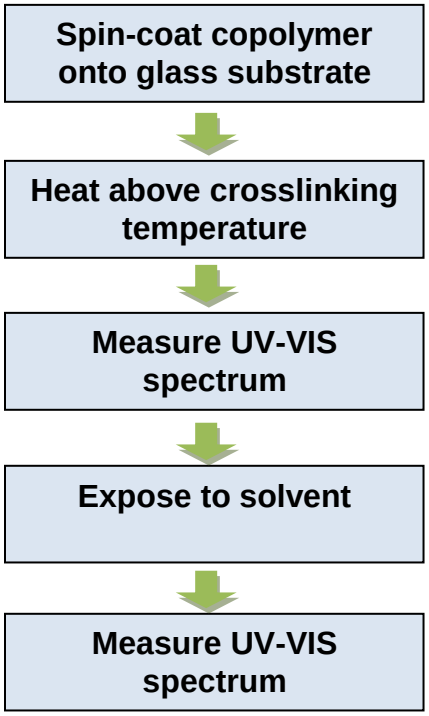
II. Thermal Crosslinking of BCB and TFVE Copolymers

Once the synthesis of the TFVE and BCB- based target materials (IC-II-32 and IC-II-30 respectively), was completed, thin films were produced from the resulting copolymers. The films were then processed under variable conditions to induce crosslinking, and the resistance of the films to solvent treatment was then evaluated. The overall process, illustrated in scheme 3 below, consisted of the following steps: 1) First the copolymer was dissolved and spin-coated unto a glass substrate. 2) The films were then heated (to 175, 200, 230, or 300 ^{o}C) for variable durations (30, 60, 120, or 240 minutes) in order to induce crosslinking (Steps 1 and 2 were performed by Carlos Zuniga). 3) Finally, the solvent resistance imparted by crosslinking was evaluated. For every film a reference UV-VIS spectrum was first taken. The film was then exposed to a solvent for variable durations (10, 30, 60, and 90 seconds), with the UV-VIS spectrum of the films being measured after every dip.

Since the absorbance is directly proportional to the concentration, by comparing the absorbance of the film after solvent exposure to the reference spectrum, one can determine how much of the initial concentration of polymer remained after the film was exposed to the solvent. It was expected that in the absence of crosslinking, no organic material would remain on the glass substrate, and consequently that the concentration of polymer detected corresponds to polymer that had been rendered insoluble by crosslinking. This assumption was verified by performing the same protocol on a control film that was not processed to induce crosslinking. Finally, by taking the ratio of the absorbance of the film after repeated exposure to solvent to the absorbance of the

reference film, an estimate for the solvent resistance of the film was obtained, which in turn was taken as an indicator of the crosslinking effectiveness.

Scheme 4



III. Rapid Thermal Processing (RTP) of BCB, TFVE Copolymers and Thermal Processing of OXT Copolymer

In a second set of studies carried out by Dr. Jassem Abdallah, the solvent resistance of the BCB, TFVE, and OXT copolymers (IC-II-30, IC-II-53, IC-II-99) was evaluated. It was attempted to induce crosslinking in the BCB and TFVE films via rapid thermal processing (RTP) treatment and in the case of the OXT copolymers by conventional thermal processing on a hotplate. The overall process, as illustrated in scheme 5 below, consisted of the following steps: 1) The copolymer was spin coated onto

a silicon wafer. In case of the OXT copolymers, blends of the copolymer and a thermal acid generator (TAG) - DPI-TPFPB (structure shown in figure 2) were used with variable TAG loadings: 0.5%, 1%, and 2%. 2) The thermal/RTP process was applied. 3) The copolymer was exposed to solvent. 4) The films were vacuum baked at eleveated temperatures. The thickness of the film was measured after each step using ellipsometry. It was expected that for films exhibiting effective solvent resistance (due to a high level of crosslinking) the thickness would not change from the first to the last step, while for films exhibiting no solvent resistance the thickness would diminish significantly. The processing conditions used to carry out the thermal/RTP processing are provided in tables I and II.

Step	**Description**
1	Spin-coating followed by 5 min drying at 90 ^{o}C
2	RTP cure at 300 ^{o}C using RTP Profile I (see Appendix B)
3	Surface washing with $CHCl_3$
4	Overnight vacuum bake at 120 ^{o}C

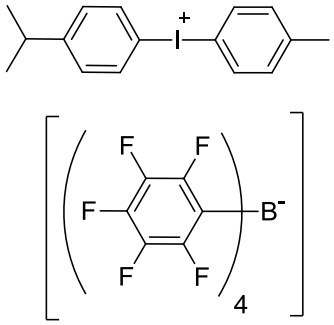
Table I: Processing Conditions for TFVE and BCB Films (IC-II-53 and IC-II-30)

Step	**Description**
1	Spin-coating followed by 10 min drying at 110 ^{o}C
2	30 min cure at 200 ^{o}C on hotplate
3	Surface washing with $CHCl_3$ followed by drying for 60 min at 60 ^{o}C
4	Overnight vacuum bake at 120 ^{o}C

Table II: Processing Conditions for OXT Films (IC-II-99)

Scheme 5


Spin-coat copolymer onto silicon wafer
Measure Film Thickness
Apply RTP Process
Measure Film Thickness
Expose to Solvent
Measure Film Thickness
Vacuum Bake at Elevated Temperature
Measure Film Thickness



4-isopropyl-4'-methyldiphenyl iodonium tetrakis(pentafluorophenyl)borate

DPI-TPFPB

Figure 2: Structure of DPI-TPFPB

RESULTS

Synthesis:

All copolymers were succesfully synthesized, purified, and characterized using ^{1}H NMR spectroscopy, gel permation chromatography (GPC), and elemental analysis. In addition, all new intermediates synthesized were characterized by ^{1}H NMR and ^{13}C NMR spectroscopy, mass spectrometry, and elemental analysis. The three copolymers contain an identical hole transporting group on one chain, but a different crosslinking group on the other: BCB in the case of IC-II-30, TFVE in the case of IC-II-32, and OXT in the case of IC-II-99 (see scheme 3). A second batch of the TFVE copolymer was synthesized and labelled IC-II-53.

Thermal Crosslinking of BCB and TFVE Copolymers

The copolymer containing the BCB group (IC-II-30) exhibited robust solvent resistance after suitable processing conditions. While the films only showed a low level of crosslinking, even after 4 hrs of heating at 200 ^{o}C, a high level of crosslinking (ca. 80%) was achieved after 4 hrs of crosslinking at 230 ^{o}C. Furthermore, even after crosslinking for 60 min at 230 ^{o}C, significant (ca. 40%) crosslinking was observed. The most promising results occurred at 300 ^{o}C, where effective crosslinking (above 90%) was obtained after 60 min.

The results of the thin-film studies for the copolymer containing the TFVE group (IC-II-32) showed no observable crosslinking at any temperature for any duration. This result was unexpected, especially in light of the fact that films processed at identical conditions containing a similar copolymer previousy synthesized in the group, containing

the same hole transport group and the same crosslinking group (connected via a different linker) showed a significant degree of crosslinking. In order to explore the possiblity that the lack of apparent crosslkining was particular to this batch of copolymer (e.g. due to a low degree of incorporation of the crosslinker), a second batch of the copolymer, IC-II-53 was prepared, which was subsequently used in the rapid thermal processing studies (see next section). The UV-VIS spectra mentioned are provided below (see Appendix A for the complete set of spectra):

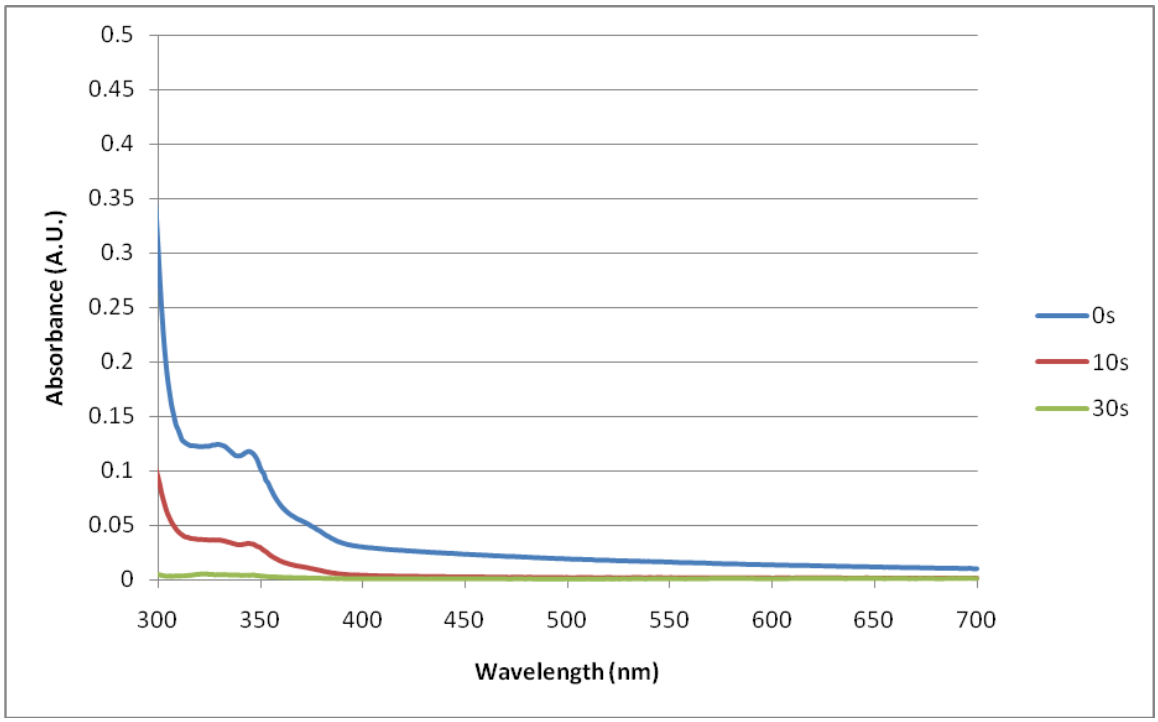


Figure 3: Absorption of Unprocessed IC-II-30 Film (Control) vs. Duration of $CHCl_3$ Wash

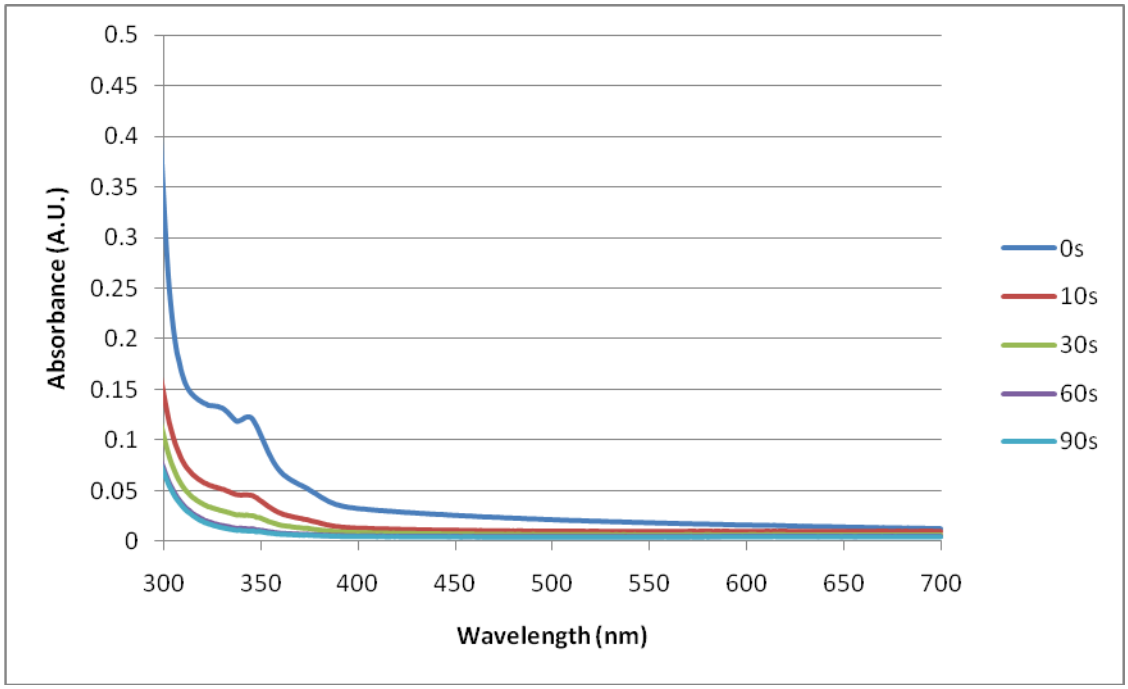


Figure 4: Absorption of IC-II-30 Film Processed at 200 ^{o}C for 240 min vs. Duration of $CHCl_3$ Wash

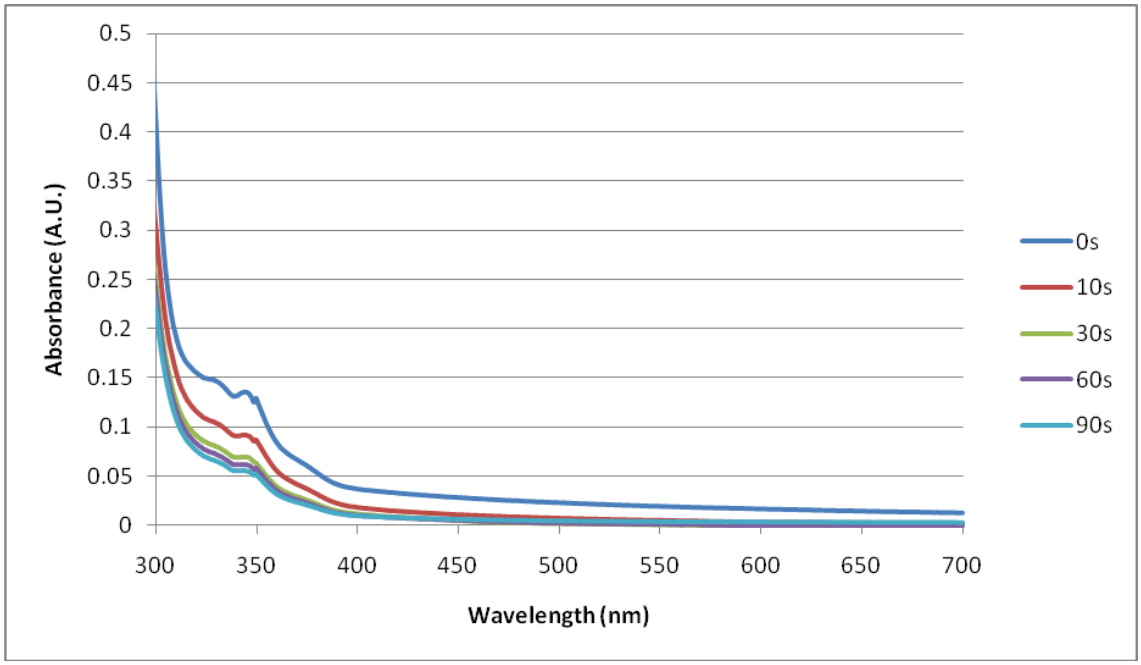


Figure 5: Absorption of IC-II-30 Film Processed at 230 ^{o}C for 60 min vs. Duration of $CHCl_3$ Wash

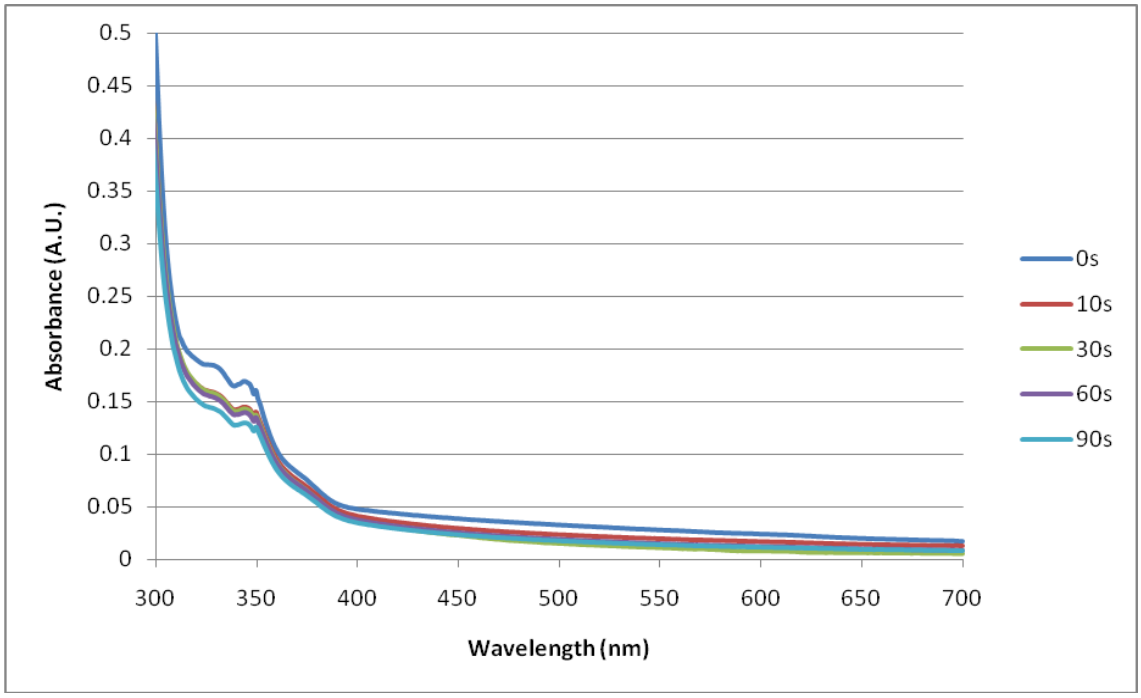


Figure 6: Absorption of IC-II-30 Film Processed at 230 ^{o}C for 240 min vs. Duration of $CHCl_3$ Wash

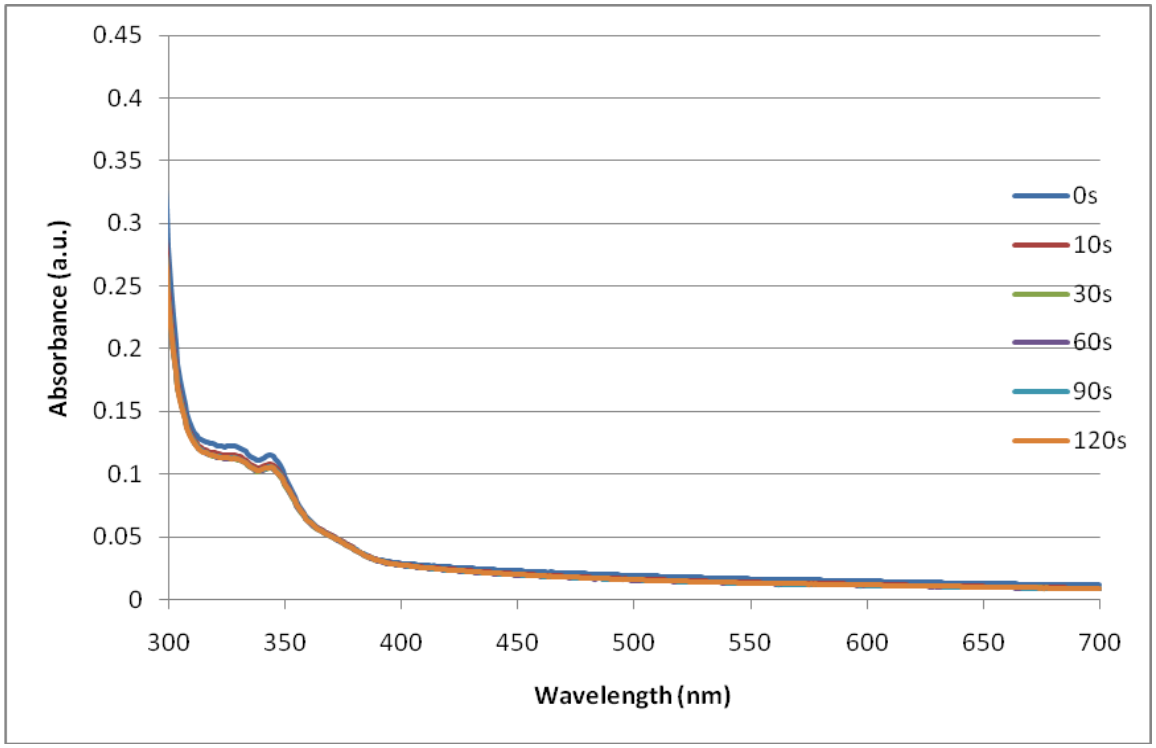


Figure 7: Absorption of IC-II-30 Film Processed at 300 ^{o}C for 60 min vs. Duration of $CHCl_3$ Wash

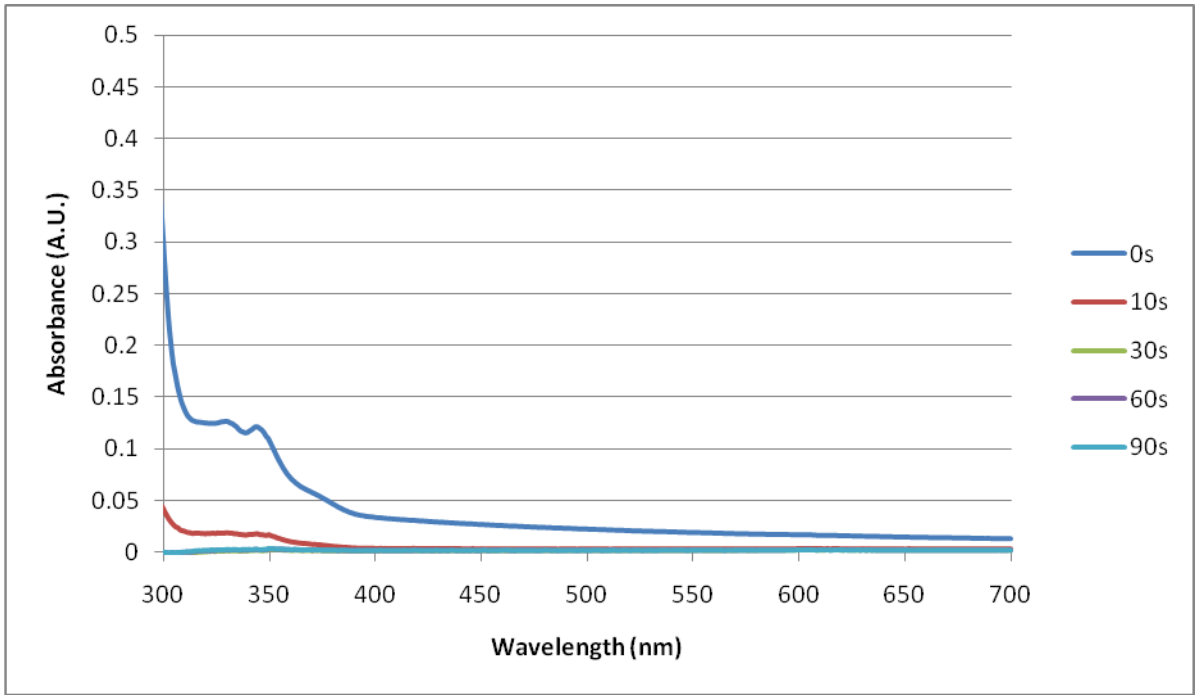


Figure 8: Absorption of Unprocessed IC-II-32 Film (Control) vs. Duration of $CHCl_3$ Wash

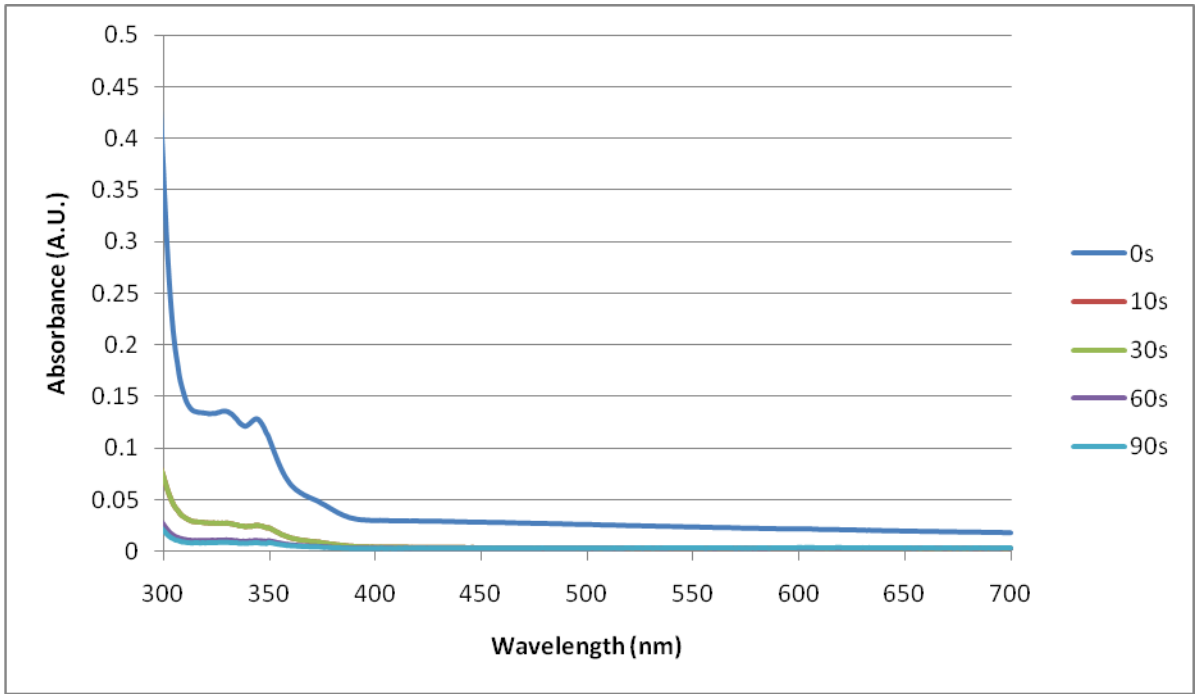


Figure 9: Absorption of IC-II-32 Film Processed at 230 ^oC for 240 min vs. Duration of $CHCl_3$ Wash

Rapid Thermal Processing (RTP) of BCB, TFVE Copolymers and Thermal Processing of OXT Copolymer

In a second set of studies, the crosslinking properties of the BCB-based copolymer (IC-II-30), the second batch of TFVE-based copolymer (IC-II-53), and the OXT based copolymer (IC-II-99) was evaluated. As was found in the UV studies, effective crosslinking was not detected for the TFVE copolymer. On the other hand, even after short processing times (30 minutes or less for both copolymers), effective crosslinking was observed both in the case of the BCB and OXT copolymers. The change of thickness in the films (as monitored by ellipsometry) after each processing step (described in Table III) are provided below:

Step	Description
1	After spin coating and drying
2	After RTP/Thermal Processing
3	After chloroform exposure
4	After overnight vacuum bake at 120 °C

Table III: Description of RTP Steps

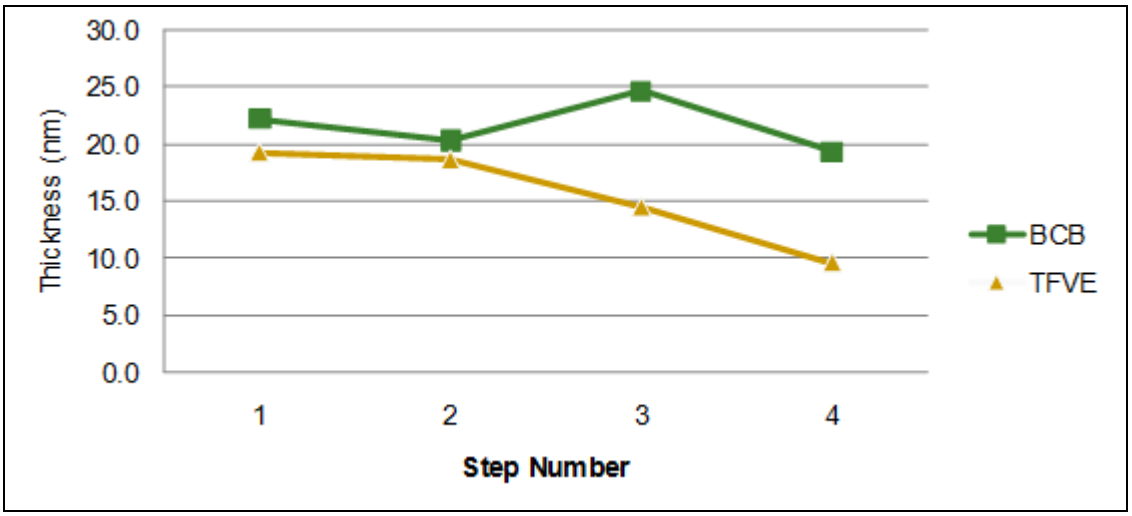


Figure 10: Ellipsometry-Based Crosslinking Evaluation of RTP Processed IC-II-30 (BCB) and IC-II-53 (TFVE) Films

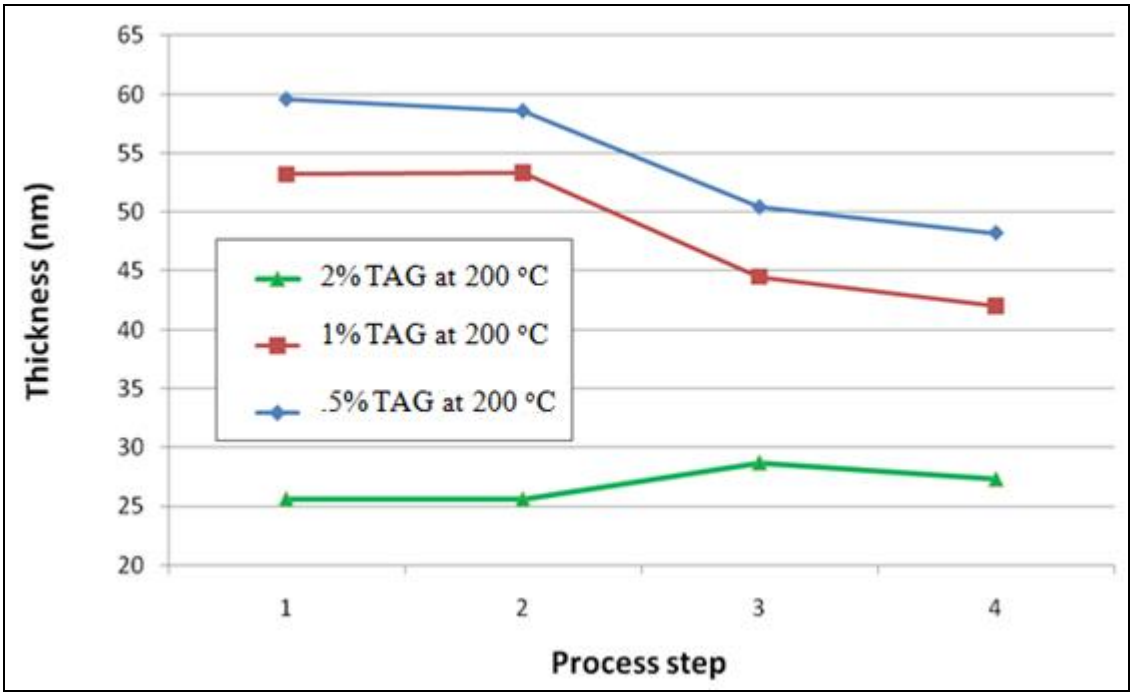


Figure 11: Ellipsometry-Based Crosslinking Evaluation of Thermally Processed IC-II-99 (OXT) Film

CONCLUSION

In conclusion, three new crosslinkable hole-transporting copolymers were prepared and their solvent resistance was evaluated. Effective crosslinking after short processing times was observed in the case of copolymers incorporating the thermally crosslinkable benzocyclobutene (BCB) and oxetane (OXT) groups, while no effective crosslinking resistance was detected for the trifluovinyl ether (TFVE) based copolymer. Materials have been transferred to the Kippelen group, where OLEDs incorporating these materials will be prepared and their optoelectronic properties will be studied.

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APPENDIX A: UV-VIS CROSSLINKING STUDIES OF IC-II-30

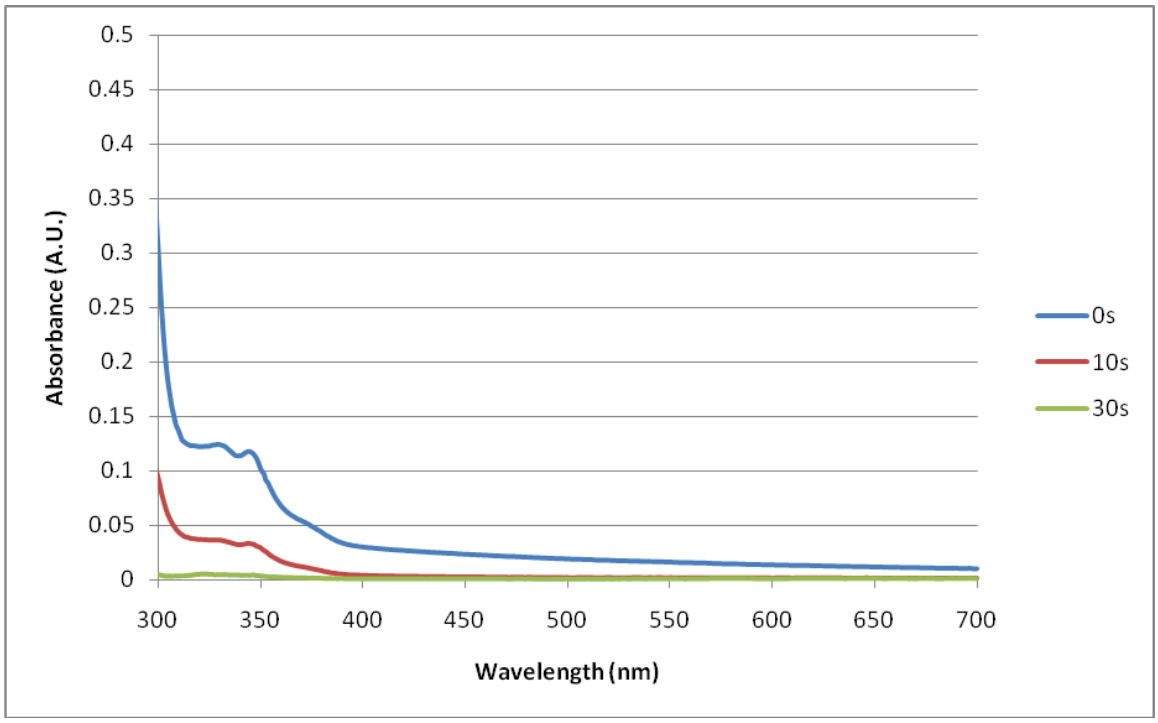

Figure A1: Absorption of Unprocessed IC-II-30 Film (Control) vs. Duration of $CHCl_3$ Wash

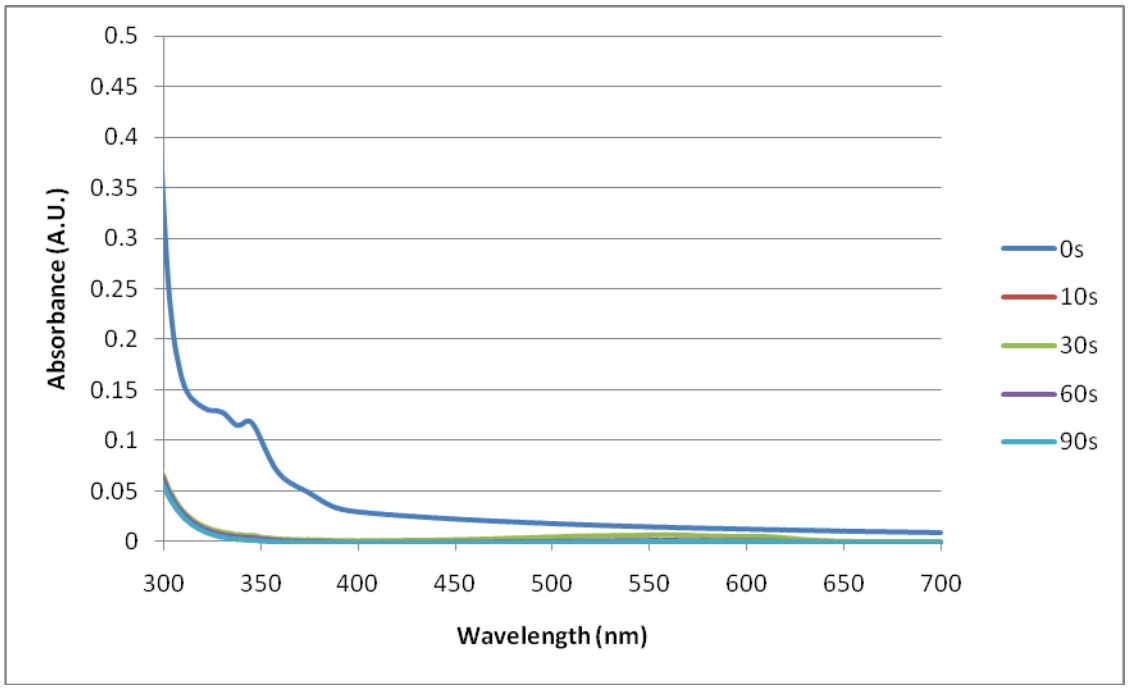

Figure A2: Absorption of IC-II-30 Film Processed at 200 ^{o}C for 30 min vs. Duration of $CHCl_3$ Wash

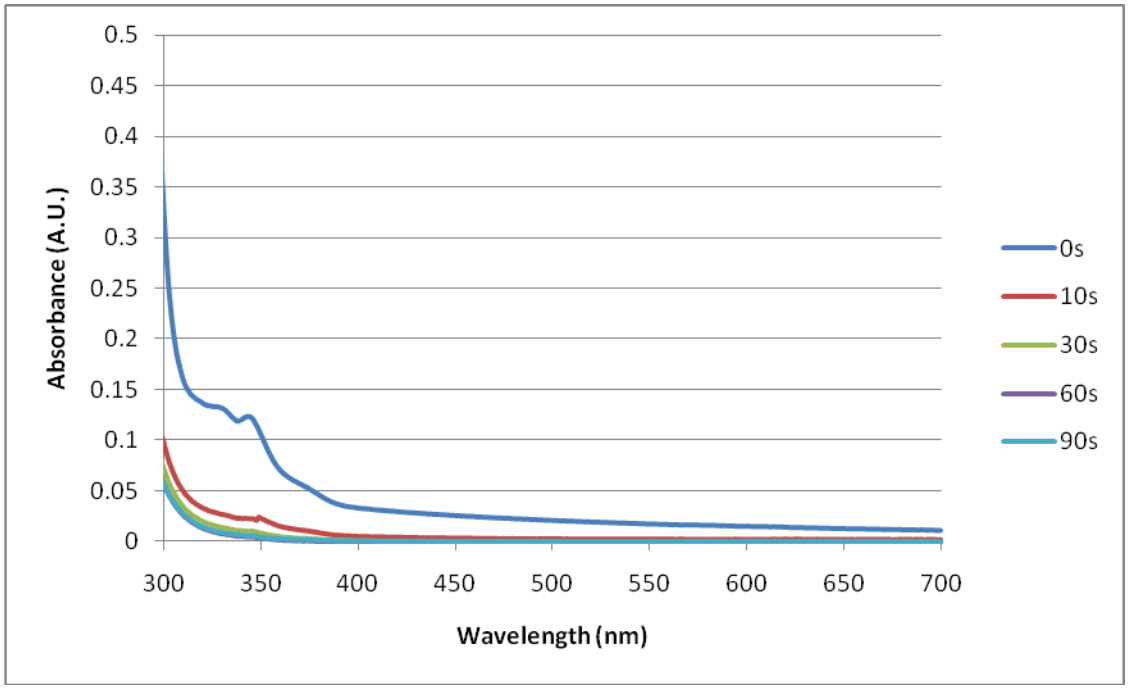

Figure A3: Absorption of IC-II-30 Film Processed at 200 ^{o}C for 60 min vs. Duration of $CHCl_3$ Wash

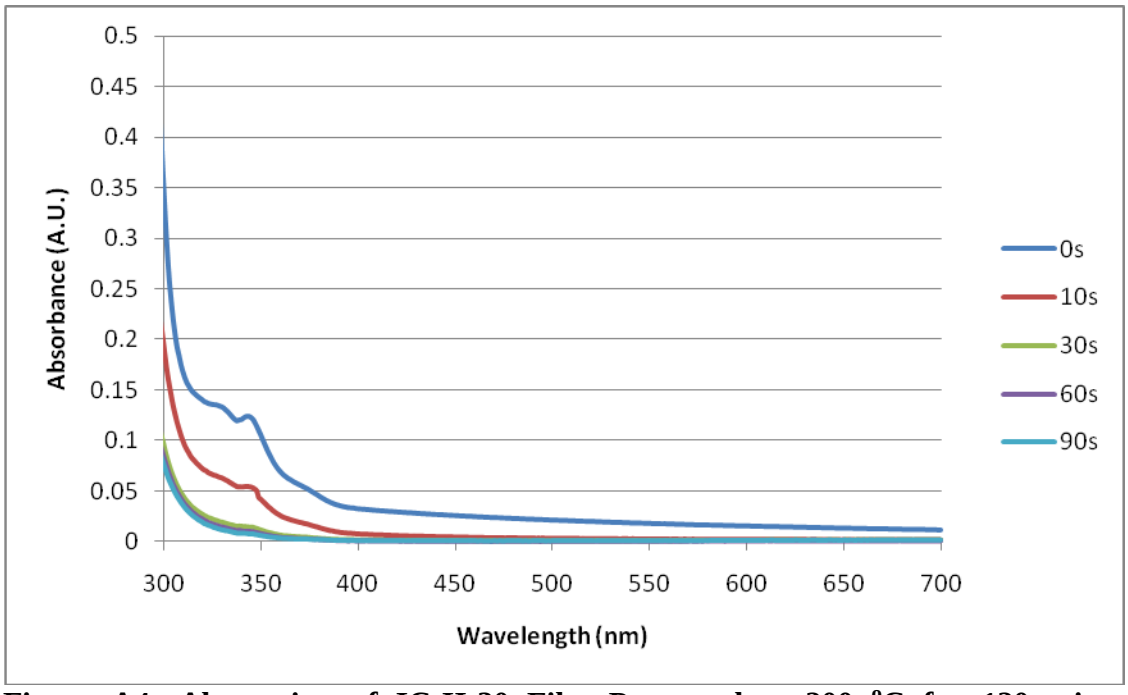

Figure A4: Absorption of IC-II-30 Film Processed at 200 ^{o}C for 120 min vs. Duration of $CHCl_3$ Wash

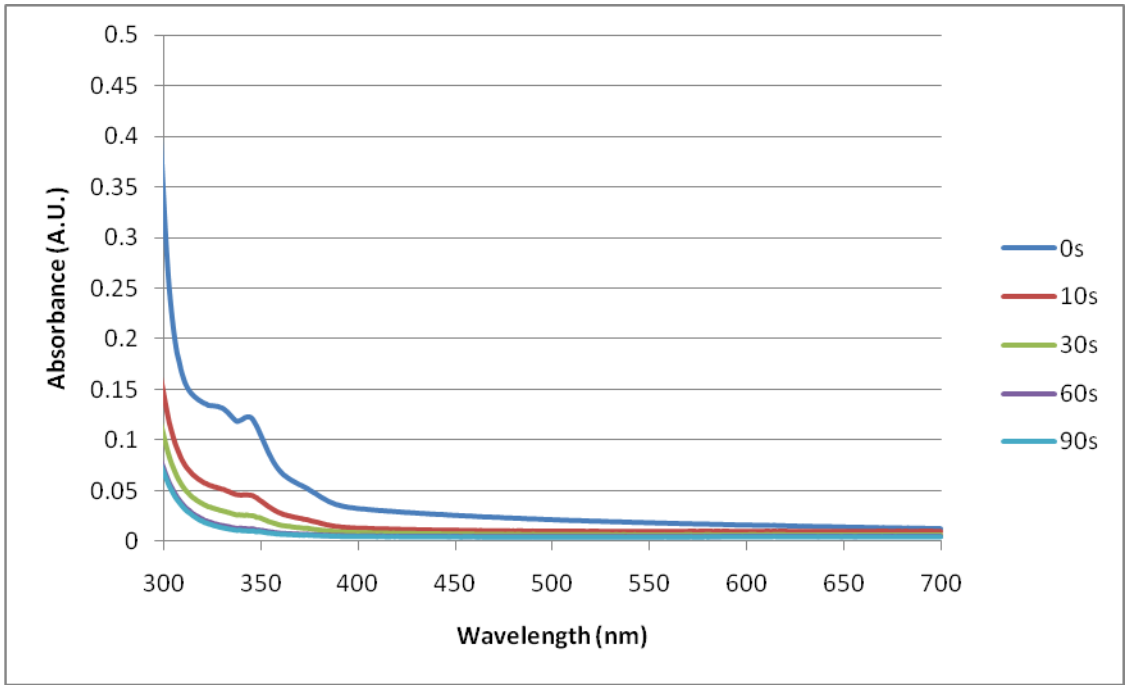

Figure A5: Absorption of IC-II-30 Film Processed at 200 ^{o}C for 240 min vs. Duration of $CHCl_3$ Wash

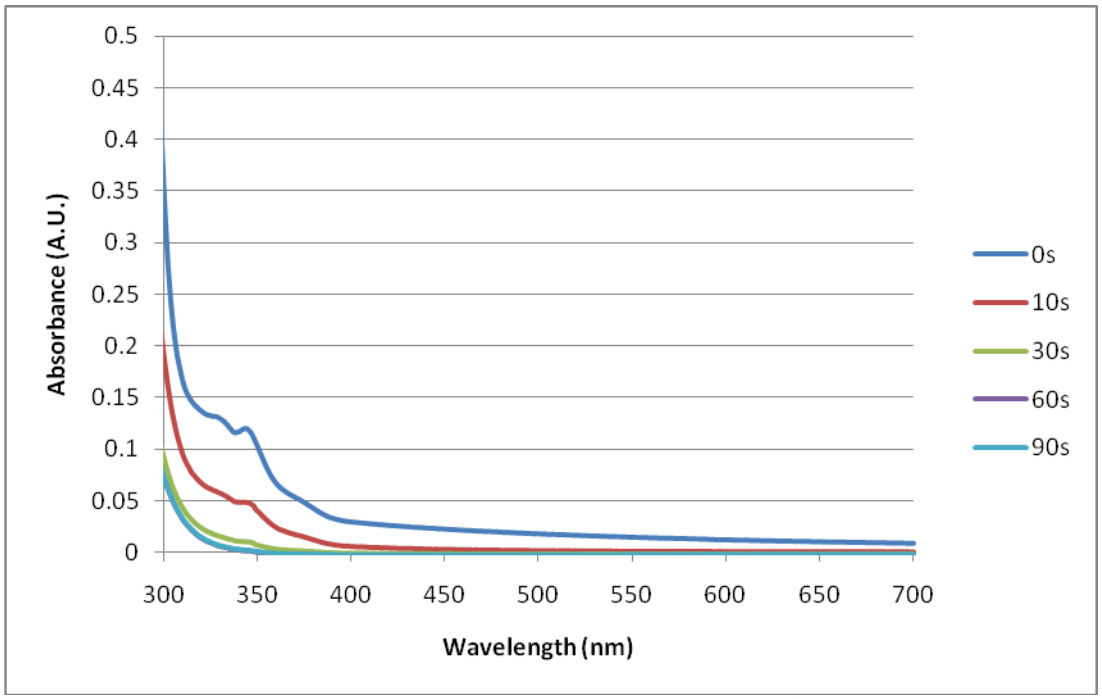

Figure A6: Absorption of IC-II-30 Film Processed at 230 ^{o}C for 30 min vs. Duration of $CHCl_3$ Wash

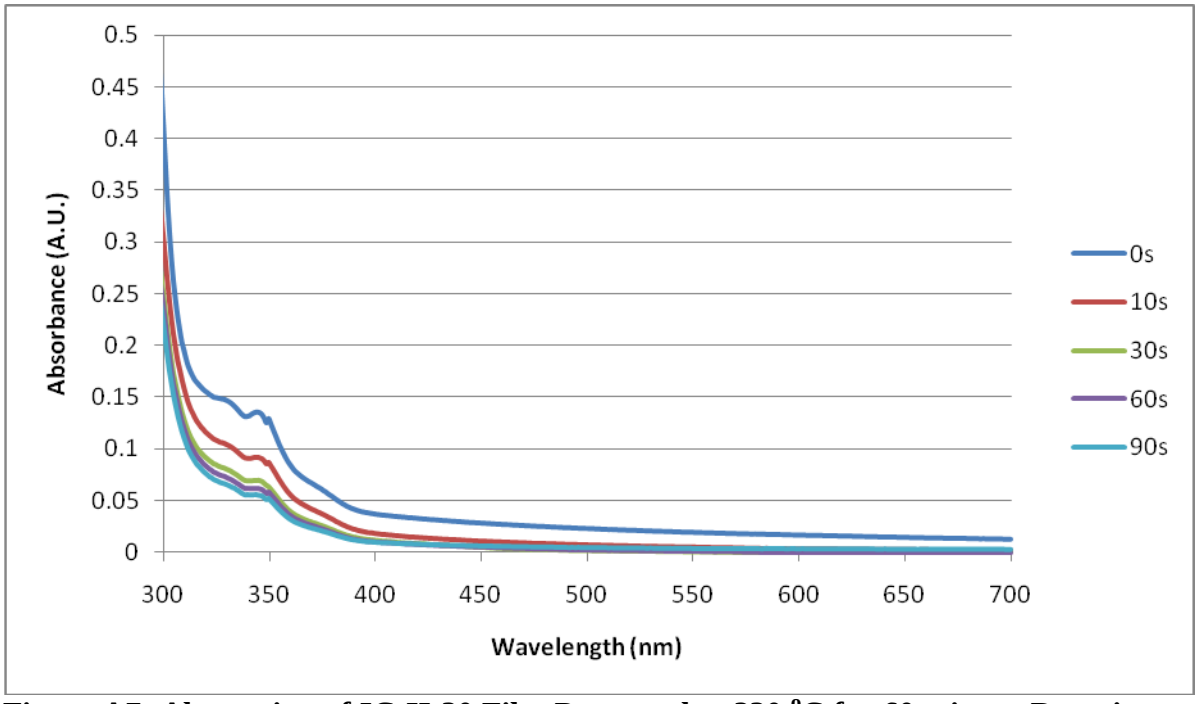

Figure A7: Absorption of IC-II-30 Film Processed at 230 ^{o}C for 60 min vs. Duration of $CHCl_3$ Wash

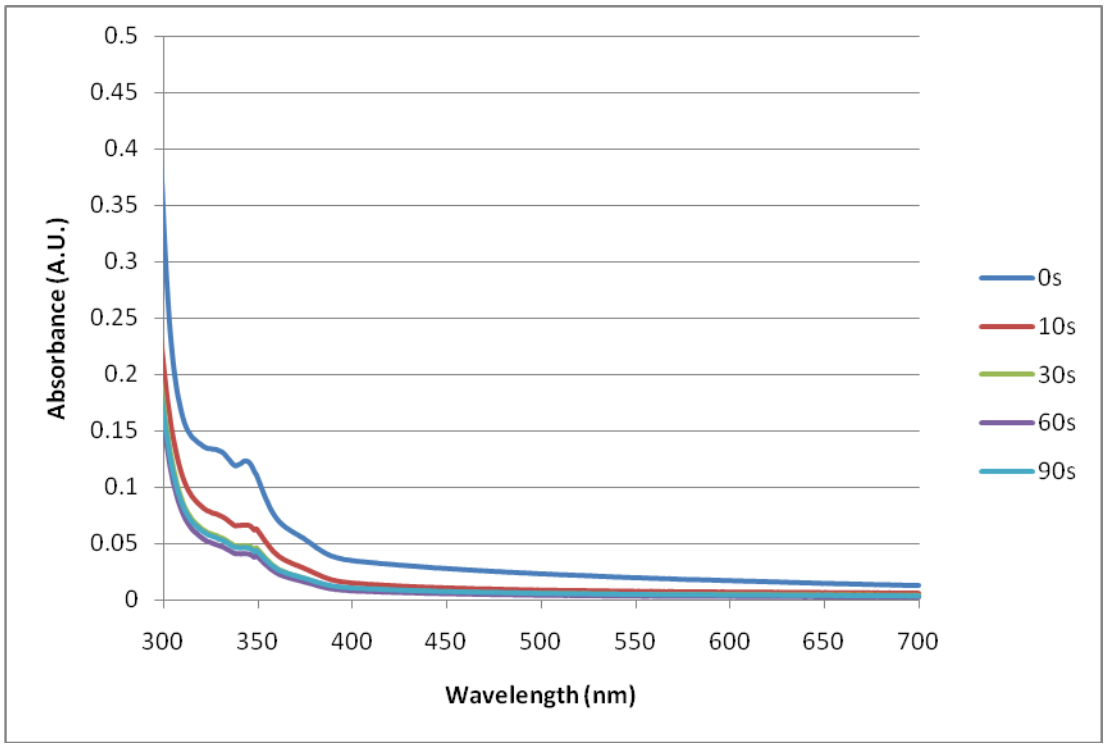

Figure A8: Absorption of IC-II-30 Film Processed at 230 ^{o}C for 120 min vs. Duration of $CHCl_3$ Wash

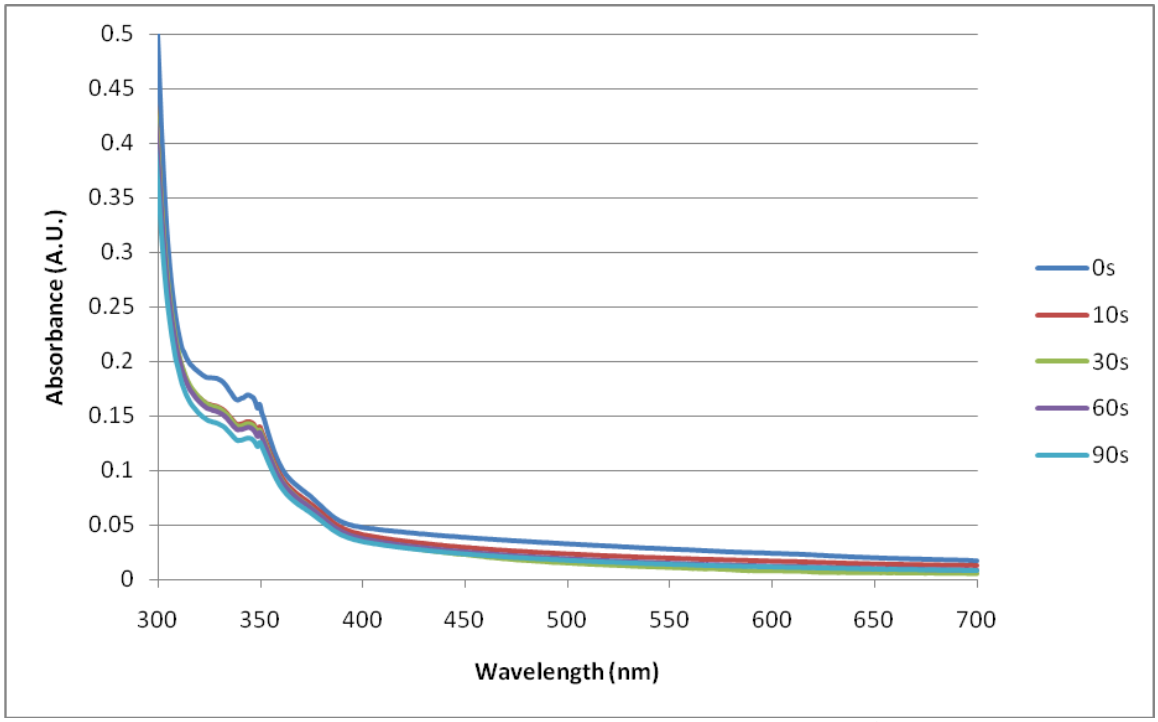

Figure A9: Absorption of IC-II-30 Film Processed at 230 ^{o}C for 240 min vs. Duration of $CHCl_3$ Wash

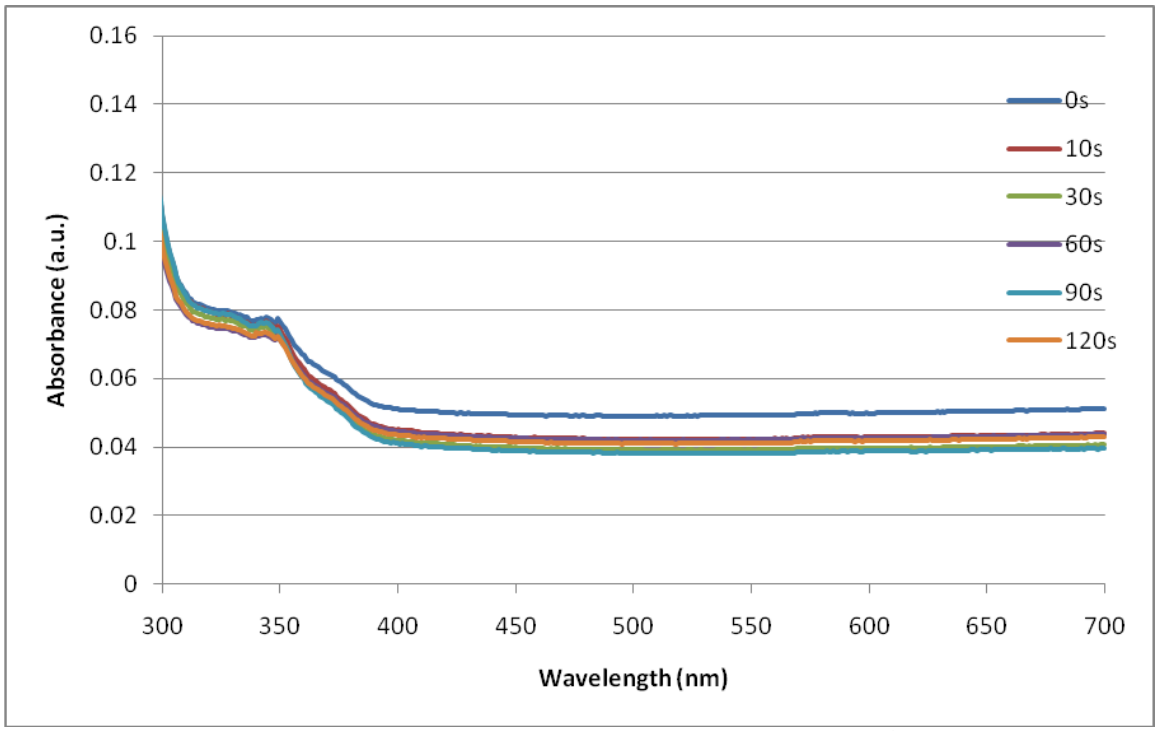

Figure A10: Absorption of IC-II-30 Film Processed at 300 ^{o}C for 30 min vs. Duration of $CHCl_3$ Wash

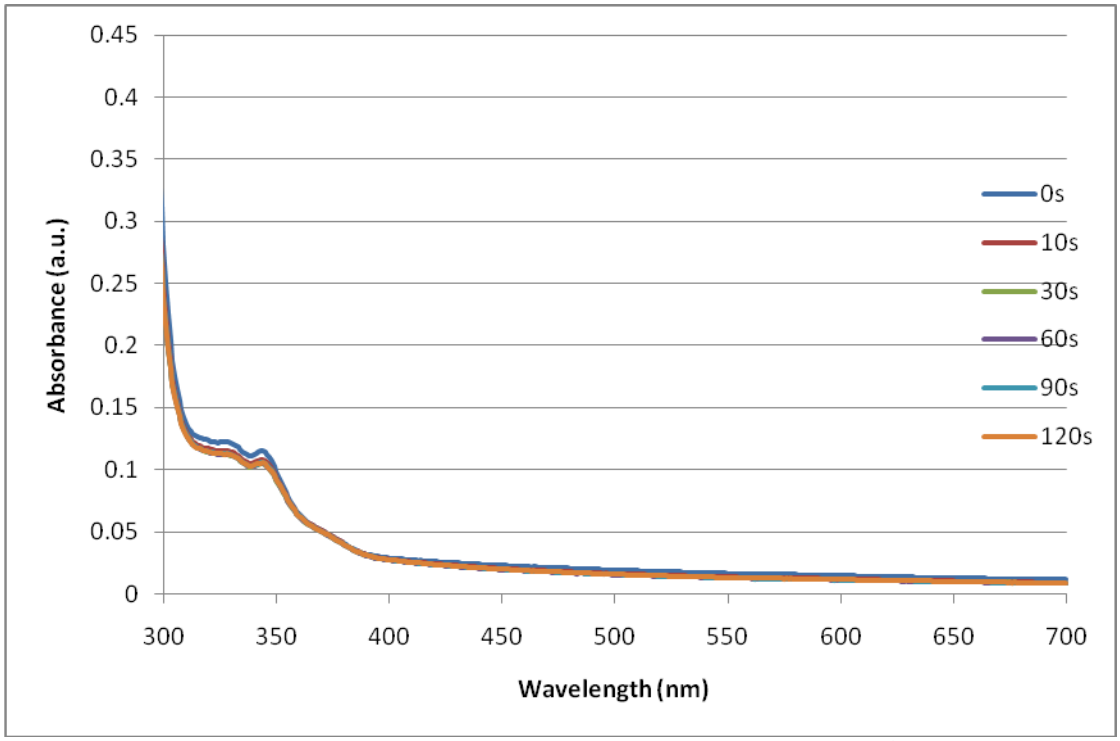

Figure A11: Absorption of IC-II-30 Film Processed at 300 ^{o}C for 60 min vs. Duration of $CHCl_3$ Wash

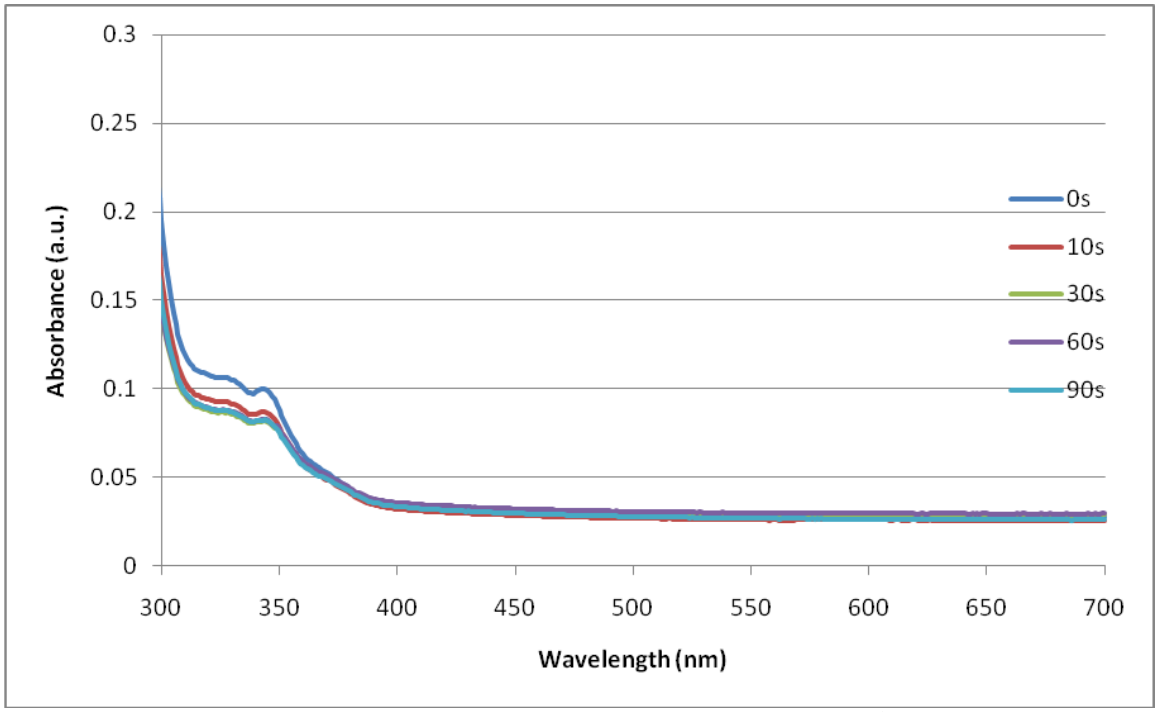

Figure A12: Absorption of IC-II-30 Film Processed at 300 ^{o}C for 120 min vs. Duration of $CHCl_3$ Wash

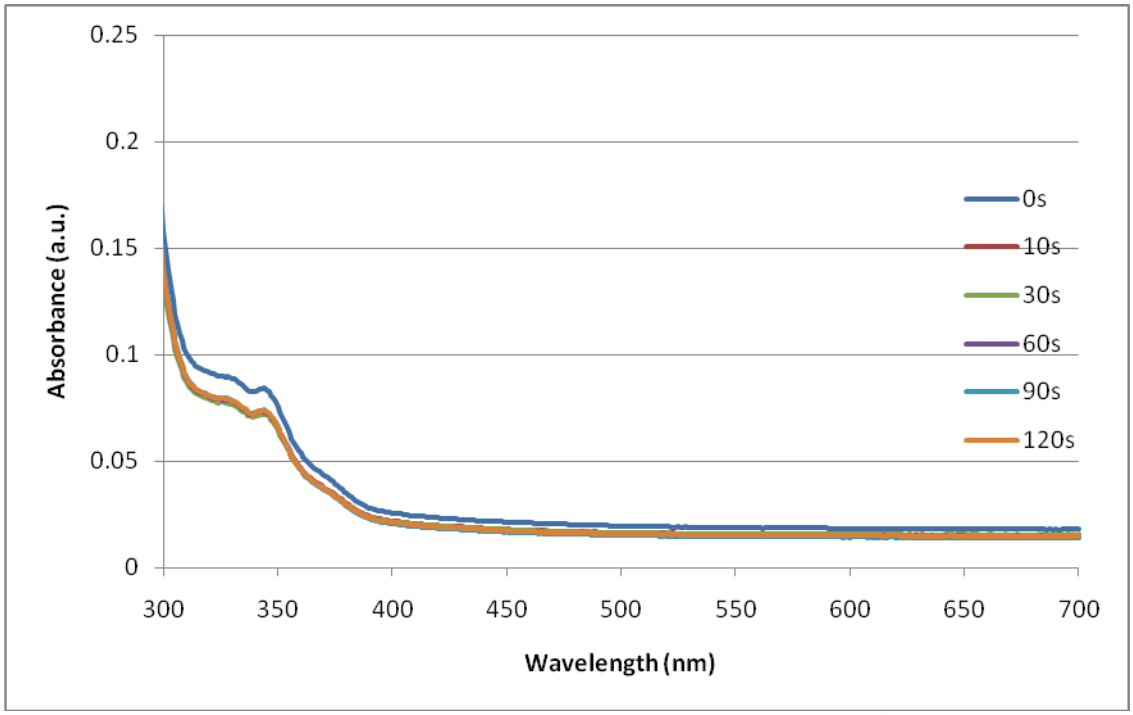

Figure A13: Absorption of IC-II-30 Film Processed at 300 ^{o}C for 240 min vs. Duration of $CHCl_3$ Wash

APPENDIX B: RTP PROFILE

RTP step	Ramp Rate	Step Time (min)	Total Time Elapsed (min)
N_2 purge at ambient	N/A	3.00	3.00
ramp 1	150^{o}C/min	1.57	4.57
ramp 2	50^{o}C/min	0.87	5.43
Dwell at 300^{o}C	N/A	5.00	10.43
cooling to 180^{o}C from 300^{o}C	N/A	2.25	12.68
cooling to 100^{o}C from 300^{o}C	N/A	9.00	19.43

Table IV: Detailed RTP Profile